SYNTHESIS AND CHARACTERIZATION OF CARBIDE-DERIVED CARBON NANOWIRES

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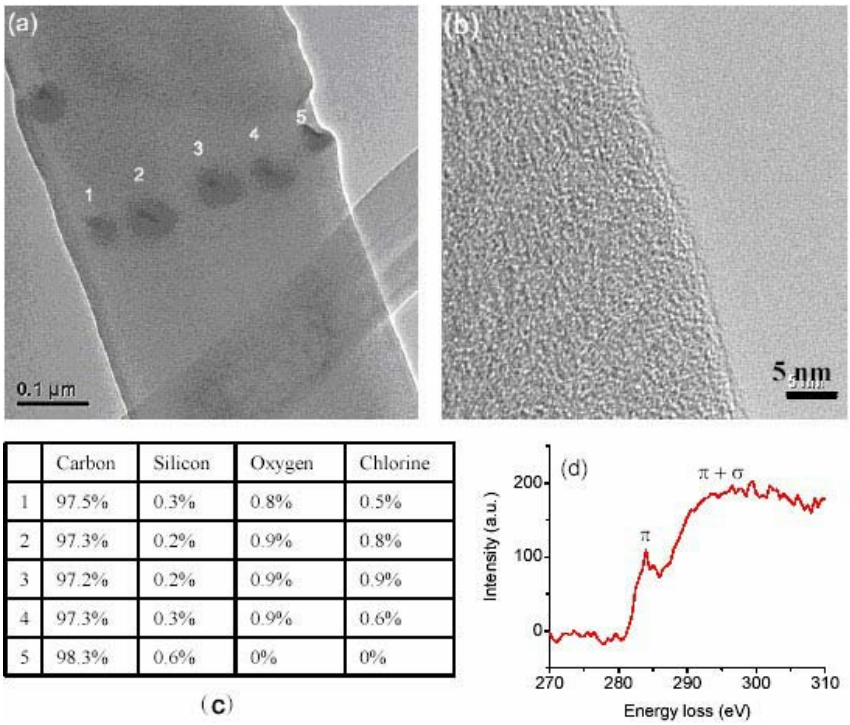
Abstract

Carbon materials such as diamond, graphite, and amorphous carbon attract attention due to their physical and chemical properties. Gogotsi et al.[1] reported the synthesis of nano- and micro-crystalline "diamond-structured" carbon films by extracting silicon from the surface region of macroscopic silicon carbide samples with chlorine-containing gases at ambient pressure and temperatures not exceeding 1,000˚C. This "carbide-derived carbon (CDC) method" has been used for the conversion of carbide to nano-crystalline diamond and graphite films on SiC.[1,2] In this work, we have used small diameter silicon carbide whiskers as the precursor and have successfully extracted Si to create carbon nanowires by the CDC method.

The β-silicon carbide whiskers (Advanced Composite Materials Corp., South Carolina) used as the precursor in this work are of high purity (99%) with 0.45–0.65 micron diameters and 5- 80 micron lengths. Our X-ray diffraction (XRD) data of the as-received whiskers proves they are β-SiC. Silicon extraction experiments have been carried out in a home-built thermal chemical vapor deposition (CVD) apparatus. The green SiC whisker precursor material was converted to a dark black product after 8 hours of the extraction reaction at 1000°C. Transmission electron microscope (TEM) images are shown in Fig. 1(a) and (b). The high-resolution TEM image and electron diffraction data (not shown) indicate that the structure is amorphous. Energy Dispersive X-ray Analysis (EDX) has been performed on the product and the data in Fig. 1(c) show >97% carbon with the remainder being oxygen, silicon and chlorine. There was no significant variation of the composition across the specimen (by EDX analysis) for the 5 spots shown in Fig. 1(a). The fraction of sp^2-bonded atoms in the specimen is ~0.88 according to EELS analysis.



	Carbon	Silicon	Oxygen	Chlorine
1	97.5%	0.3%	0.8%	0.5%
2	97.3%	0.2%	0.9%	0.8%
3	97.2%	0.2%	0.9%	0.9%
4	97.3%	0.3%	0.9%	0.6%
5	98.3%	0.6%	0%	0%

(c)

Figure 1. (a) Low-magnification TEM image of the product nanowire; (b) High-magnification TEM image of the product nanowire; (c) atomic percentages assigned by the Hitachi HF-2000 software from the five spots in (a); (d) EELS spectrum taken with the Hitachi HF-2000 at the edge of the product nanowire.

Materials with high specific surface area and controlled pore size distribution have potential applications as molecular sieves, and for gas storage, catalysis, and use as absorbents, battery electrodes, supercapacitors, water/air filters, and medical devices. The TEM images of these carbon nanowires suggest a nanoporous structure. The surface area and pore size have been measured with a *Micromeritics ASAP 2010 Accelerated Surface Area and Porosimetry* analyzer. Isotherms of the adsorption of nitrogen were recorded at -195.8 ° C. The Langmuir surface area obtained in one measurement on 100 mg of material was 1658 m^2/g and the surface area analyzed by the micropore analysis method for the same sample was 1978 m^2/g: *these two surface areas show we have synthesized a new ultra-high surface area material composed primarily of carbon and in nanowire form*. The pore size distribution was calculated by the Horvath-Kawazoe method and the median pore radius is 0.57 nm.

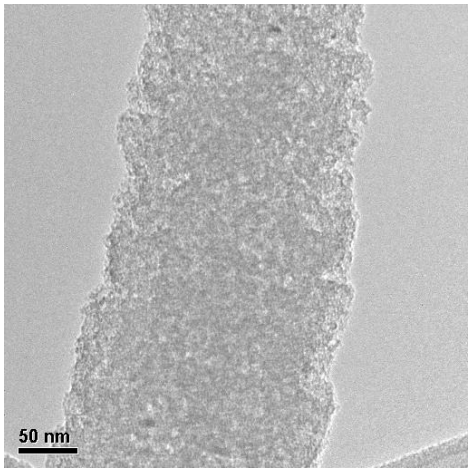


Figure 2. TEM image of porous carbon NW derived from silicon carbide.

In summary, we have converted silicon carbide whiskers to carbon nanowires by the CDC method and the analysis indicates that the carbon nanowires have very high surface area and a nano-porous structure. The EELS results show primarily sp^2 C-C bonding. We are currently trying to enhance the fraction of sp^3 bonding in an attempt to make diamond-like, or even single crystal diamond, nanowires.

Acknowledgement

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REFERENCES

1. Gogotsi, Y., et al., "Conversion of silicon carbide to crystalline diamond-structured carbon at ambient pressure", *Nature*, Vol. 411, No. 6835, 283 (2001).
2. Gogotsi, Y. G., Jeon, I.-D. & McNallan, M. J. "Carbon coatings on silicon carbide by reaction with chlorine-containing gases", *J. Mater. Chem.,* Vol. **7**, No. 9, 1841 (1997).